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## (54) METHOD OF PRODUCING FOAMED CERAMIC MATERIAL

(71) We, GOSUDARSTVENNY NAUCHNO-ISSLEDOVATELSKY INSTITUT STROITELNO-KERAMIKI, of Moskovskaya oblast, g. Zheleznodorozhny, Union of Soviet Socialist Republics, a Body Corporate of the Union of Soviet Socialist Republics, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of producing foamed ceramic materials.

There are, at the present time, widely known methods of producing foamed ceramic materials from a foam formed by the mechanical foaming of an aqueous suspension of a non-plastic material with the resinous saponin extract from soap root (as foaming agent) and a structure stabiliser. Typical structure stabilisers include gypsum, alum and sawdust (cf. the Journal "Refractories", No. 2, 1959, Metallurgizdat, Moscow, article by I. Ya. Guzman and D. N. Polubojarinov, "Lightweight Refractories from Aluminium Oxide"). From the foam composition thus obtained are made articles which are then dried and fired.

The principle disadvantage of the known methods is the very low mechanical strength of the foam composition obtained. This complicates subsequent manufacturing processes on the articles and makes it impossible to obtain articles having a density less than 0.5 g/cm<sup>3</sup> or having large dimensions. Because of the low mechanical strength of

the foam composition, the first-stage drying of articles made from it has to be carried out in moulds. This causes cracking of the articles due to non-uniform shrinkage and prolongs the drying period.

It has now been found that certain of the disadvantages of the known process may be overcome by the provision of a method of making a foamed ceramic material, which comprises preparing a stabilised foamed composition comprising an aqueous suspension of a non-plastic ceramic material, a foaming agent, gelatin and formalin, cooling said stabilised foamed composition to a temperature not greater than 0°C and maintaining said stabilised foamed composition at a temperature not greater than 0°C until it has solidified. Gelatin acts as the foam stabiliser. When the composition is cooled down, a reaction between formalin and gelatin takes place, as a result of which the foamed composition becomes mechanically stronger. In order to obtain the best results, it is preferred to use the gelatin in an amount of from 1 to 10% and the formalin (a 40% aqueous solution of formaldehyde) in an amount of from 0.5 to 5%, based on the weight of the starting non-plastic material.

The non-plastic (non-clay) ceramic material, which, in contrast to plastic materials, such as clayey materials, does not become plastic when mixed with water, may be, for example, aluminium oxide, zirconium oxide, beryllium oxide, titanium oxide, cordierite, celsian, titanates of barium, cal-

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cium and strontium, metal carbides or metal borides.

An aqueous suspension of this non-plastic material is mixed with a foaming agent, such as resinous saponin extract from soap root, and with a colloidal solution of gelatin, used as the foam stabiliser. This suspension is then foamed in a mixer. The foaming operation may be effected in mixers conventionally employed for the preparation of foamed ceramic materials. The time of foaming in the mixer depends upon the type of mixer used, the density of the foamed composition and the type of non-plastic ceramic material; it is usually from 10 to 30 minutes.

Having foamed the suspension to the desired density, formalin is added to the foamed composition. Following this, articles can be formed from the foamed composition by casting the composition into metallic moulds. The articles are cooled to a temperature not exceeding 0°C and are maintained at such a temperature until the foamed composition solidifies. The time required for this solidification depends upon the actual cooling temperature and on the size of the articles made. Under these conditions, interaction occurs between the formaldehyde and the gelatin which strengthens the foamed composition. When the solidification is completed, the articles are removed from their moulds and dried by high-frequency heating. The drying time depends upon the size and density of the article, generally being 2-3 hours.

After drying, the articles are fired. The firing temperature depends upon the type of starting materials used. Firing may be accomplished by conventional means, since the strengthening agents used in the present invention do not have any noticeable effect on the process.

The invention is now further illustrated with reference to the following Examples, which show various embodiments of the present invention. The mixer used for foaming in each of these Examples was provided with a horizontal rotating shaft having secured thereto in the horizontal direction metal spokes of 1.5 mm. diameter.

#### *Example 1*

An aqueous suspension as calcined aluminium oxide having a water content of 50% was prepared. A colloidal solution of gelatin and a 1% extract of resinous saponin from soap root were introduced into the suspension. The gelatin was used in an amount of 4% based on the weight of the aluminium oxide used and the resinous saponin extract in an amount of 12.5% based on the weight of aluminium oxide.

The suspension thus obtained was foamed in the mixer until its density reached 0.2

g/cm<sup>3</sup>. Formalin was then introduced into the foamed composition. The formalin was taken in an amount of 1.6% based on the weight of aluminium oxide.

The foamed composition was cast in metal moulds having the dimensions: 300×160×120 mm., which were placed in a freezing chamber kept at a temperature of from -10 to -15°C until the foamed composition had solidified. After 20 hours, the articles were taken out of the freezing chamber, removed from the moulds and dried for 2 hours by means of a high-frequency heater.

The density of the articles after drying was 0.14 g/cm<sup>3</sup>.

The dried articles were fired at a temperature of 1,650°C. The density after firing was approximately 0.3 g/cm<sup>3</sup>.

The material thus obtained had a compressive strength of about 20 kg/cm<sup>2</sup>; its thermal conductivity was 0.316 kcal/m.hr.deg. The minimum dimension of the pores was 0.27 mm.; the maximum dimension, 0.52 mm; and the average dimension, 0.4 mm.

#### *Example 2*

An aqueous suspension of fused quartz having a water content of 38% was prepared. A colloidal solution of gelatin and an extract of resinous saponin from soap root were introduced into the suspension. The gelatin was used in an amount of 2.4% based on the weight of fused quartz, and the resinous saponin extract was used in an amount of 9%, also based on the weight of fused quartz. The suspension so obtained was foamed in the mixer until its density reached 0.6 g/cm<sup>3</sup>. Formalin was then added to the foamed composition, in an amount of 1.0%, based on the weight of fused quartz.

The foamed composition was then cast in metal moulds having the dimensions 220×160×100 mm., which were placed in a freezing chamber kept at a temperature of from -10 to -15°C until the foamed composition had solidified. After 20 hours, the articles were taken out of the chamber, removed from the moulds, and dried for 3 hours by means of a high-frequency heater. The density of the articles after drying was 0.42 g/cm<sup>3</sup>. The dried articles were fired at a temperature of 1,200°C, and the density after firing was approximately 0.6 g/cm<sup>3</sup>.

#### *Example 3*

An aqueous suspension of zircon ( $ZrSiO_4$ ) having a water content of 35% was prepared. A colloidal solution of gelatin and an extract of resinous saponin from soap root were introduced into the suspension. The gelatin was added in an amount of

2.2%, based on the weight of zircon and the resinous saponin extract was added in an amount of 8.1%, based on the weight of zircon used. The suspension so obtained 5 was foamed in the mixer until its density reached 0.6 g/cm<sup>3</sup>. Formalin was then added to the foamed composition, in an amount of 0.9%, based on the weight of zircon.

10 The foamed composition thus obtained was cast into cylindrical metal moulds having the following dimensions: external diameter, 360 mm.; internal diameter, 100 mm.; and height, 250 mm. The moulds 15 were placed in a freezing chamber maintained at a temperature of from -10 to -15°C until the composition had solidified. After 20 hours, the articles were taken out of the chamber, removed from the moulds, 20 and dried for 3 hours by means of a high-frequency heater.

The density of the articles after drying 25 was 0.5 g/cm<sup>3</sup>. The dried articles were fired at a temperature of 1,250°C, the density after firing being approximately 0.7 g/cm<sup>3</sup>.

Although the invention has been particularly described with reference to the use of resinous saponin extract from soap root, it 30 will be appreciated that any conventional foaming agent may be used.

#### WHAT WE CLAIM IS:—

1. A method of making a foamed ceramic material, which comprises preparing a 35 stabilised foamed composition comprising an aqueous suspension of a non-plastic cera-

mic material, a foaming agent, gelatin and formaldehyde; cooling said stabilised foamed composition to a temperature not greater than 0°C and maintaining said stabilised foamed composition at a temperature not greater than 0°C until it has solidified.

2. A method according to claim 1, in which gelatin is used in an amount of from 1 to 10% and formalin is used in an amount of from 0.5 to 5%, based on the weight of said non-plastic ceramic material.

3. A method according to claim 1 or claim 2, in which said foaming agent is resinous saponin extract from soap root.

4. A method according to any one of claims 1, 2 and 3, in which said non-plastic ceramic material is aluminium oxide, zirconium oxide, beryllium oxide, titanium oxide, cordierite, celsian, a titanate of barium, calcium or strontium, a metal carbide or a metal boride.

5. A method according to claim 1, substantially as hereinbefore described.

6. A foamed ceramic material when prepared by the method of any one of the preceding claims.

7. A method in which the foamed ceramic material of claim 6 is dried and fired.

8. A method according to claim 7, substantially as hereinbefore described.

9. A dried and fired foamed ceramic material when prepared by the method of claim 7 or claim 8.

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